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PROCESS - PLATING - CRACK FREE CHROMIUM, CF-500, PHYSICAL AND CHEMICAL PROPERTIES, EVALUATION OF

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PROCESS - PLATING - CRACK-FREE CHROMIUM, CF-500, PHYSICAL AND CHEMICAL PROPERTIES, EVALUATION OF

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The tests described in this report were conducted between 10 July 1959 and 17 March 1961.

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PROCESS - PLATING - CRACK-FREE CHROMIUM, CF-500, PHYSICAL AND CHEMICAL PROPERTIES, EVALUATION OF

PURPOSE:

Conventional chromium electroplates as applied to meet Specification QQ-C-320 has no salt spray test requirements. This is because conventional chromium plate, sometimes called engineering chromium or industrial hard chromium contains microcracks. These microcracks allow corrosive media to penetrate the plating to basis metal causing corrosion. Because the electroplate and basis metal are dissimilar metals the steel basis metal used in most applications becomes anodic with an increased corrosion rate.

Crack-free 500 (CF-500) chromium is a new development by Metal and Thermit Corporation reported to contain certain stress relief additives which allows chromium to be electrodeposited in a crack free condition. The CF-500 is also reported to be softer, more ductile and more adherent. The purpose of this test is to determine the properties of CF-500 chromium.

SUMMARY:

Crack-free 500 chromium and conventional chromium plated 4130 and Thermold A steel, a H-11 type steel similar to Vascojet 1000, test specimens were plated and compared in various tests. The tests were (1) crack detection, (2) visual observations, (3) adhesion, (4) hardness, (5) salt spray accelerated corrosion tests, (6) sustained load tests and (7) effects of various bake-out conditions on hydrogen content in plated Thermold A steel.

The two types of chromium were comparable in appearance and adhesion but CF-500 chromium was slightly harder and considerably more corrosion resistant in salt spray tests. It was also determined that increasing the hydrogen relief bake-out conditions produced adequate sustained load strength of CF-500 chromium plated high heat treat steel. Hydrogen analysis test results on specimens with one or more unplated edges showed the hydrogen content was reduced by increasing the bake-out time and temperature. Adequate hydrogen embrittlement relief was obtained by baking at 500°F for 23 hours.

PROCESS - PLATING - CRACK-FREE CHROMIUM, CF-500, PHYSICAL AND CHEMICAL PROPERTIES, EVALUATION OF

OBJECT:

1. To investigate the chemical and physical properties of CF-500 chromium electroplates as compared with conventional chromium electroplates.

2. To investigate the extent of hydrogen embrittlement occurring and methods of hydrogen relief for chromium plated high heat treat steel.

SPECIMENS, MATERIALS AND EQUIPMENT:

I. Specimens

Item	Use	Source
A. 2 pieces of 4130 Steel .040" x 1" x 5"	basis metal for crack detection test	Bethlehem Steel Co. Bethlehem, Pa.
B. 2 pieces of 4130 steel .040" x 1" x 5"	basis metal for visual observation	Bethlehem Steel Co. Bethlehem, Pa.
C. 6 pieces of 4130 steel .040" x 1" x 5"	basis metal for bend adhesion	Bethlehem Steel Co. Bethlehem, Pa.
D. 2 pieces of 4130 steel .040" x 1" x 5"	basis metal for hardness test	Bethlehem Steel Co. Bethlehem, Pa.
E. 28 pieces of 4130 steel .040" x 1" x 5"	basis metal for salt spray tests	Bethlehem Steel Co. Bethlehem, Pa.
F. 2 pieces Thermold A steel .055" x 1" x 8"	basis metal for standard tensile specimen	Universal-Cyclops Steel Company Titusville and Bridgeville, Pa.

	Item	Use	Source
G.	44 pieces of Thermold A steel .055" x 1.25" x 8"	basis metal for notched tensile and sustained load test specimens.	Universal-Cyclops Steel Company Titusville and Bridgeville, Pa.
H.	3 pieces Thermold A steel .055" x .25" x 6"	basis metal specimen for hydrogen pick-up determinations	Universal-Cyclops Steel Company Titusville and Bridgeville, Pa.

II. Materials:

	Item	Use	Source
A.	125 pounds of Crack-free 500 Chromium	ingredient for Crack-free 500 chromium plating	Metal & Thermit Corp. Rahway, N. J.
B.	100 pounds of Chromic Acid Flake	ingredient for 53 oz/gal conventional chromium plating solution	Mutual Chromium Chem. Baltimore, Md.
C.	100 pounds of Oakite 190	anodic electrocleaner prior to plating	Oakite Products, Inc. 52 H. Rector St. New York 6, N. Y.

III. Equipment:

	Item	Use	Source
A.	vapor degreaser	preplate cleaning of all basis metal	manufactured by Dept. 36
B.	electroplating test fixture XJ-91602	anodic cleaning, pickling and plating of specimens	manufactured by tooling department
C.	Bausch & Lomb sterioscopic microscope	visual examination of specimens	W. H. Curtin Co. P.O. Box 5304 Dallas, Texas
D.	salt spray test cabinet type C-Al	porosity (salt spray) tests	Industrial Pump & Filter Mfg. Co., Chicago, Ill.
E.	Tukon hardness tester with Knoop Indenter	hardness tests of electroplates	Wilson Mechanical Instruments Bridgeport, Conn.

	Item	Usage	Source
F.	Research Metallograph	examination of specimens for microcracks	Bausche & Lomb Co., Rochester, N. Y.
G.	60,000 pound Baldwin universal test machine with microformer extensometer and MA-1 stress strain recorder	tensile tests	Baldwin Lima Hamilton Company Waltham, Mass.
H.	12,000 pound Arcweld creep-rupture machine	sustained load tests	Arcweld Mfg. Company Grove City, Pa.
I.	375-1000°F draft oven	application of various bake-out conditions	Blue M. Electric Co. Blue Island, Ill.
J.	vacuum hydrogen determinator	analysis of specimens for hydrogen content	National Research Corp. Newton Highland 61, Mass.
K.	cut-off saw	cutting specimens after sustained load tests	Precision Scientific Company Chicago, Ill.

PROCEDURES:

I. Application procedures for conventional chromium and Crack-free 500 chromium test finishes to 4130 and Thermold A steel.

All basis metal specimens were wiped with cheesecloth moistened with methyl ethyl ketone. They were then vapor degreased in trichloroethylene. Specimens were anodic cleaned in Oakite 190, (6 oz/gal), 160-180°F at 6 volts for 3 minutes. Thermold A steel specimens were made the anode first in the chromium plating solution for one minute at 6 volts and then quickly switched over to cathodic arrangement for chromium plate. Specimens of 4130 steel were pickled in 6 normal hydrochloric acid at room temperature for approximately two minutes just prior to plating.

The conventional chromium bath used was made up of 53 oz/gal. chromic acid flake and o.53 oz/gal. sulfuric acid catalyst. The operating temperature was 130°F ±5°F while the plating current was 2 amps/square inch. Crack-free 500 chromium plating was performed according to vendor instructions as follows:

A. The concentration of CF-500 material was 44 oz/gal and the operating temperature was 150°F ±2°F.

B. Plating current density was 2 amps/square inch.

C. The new solution was dummied with copper cathode at 6 volts for 4 hours.

D. Ordinary chromium plating anodes of tin-lead alloy were used and the solution was contained in glass.

II. Test Procedures

A. Visual Inspection

During preparation of specimens for salt spray, crack detection, sustained load, hardness etc., the various specimens were examined for pits, blisters, excessive edge build-up and other possible imperfections.

B. Adhesion

Specimens were bent repeatedly through an angle of 180°F on a diameter equal to the thickness of the specimen until fracture of the basis metal. The fracture area was then examined at 7X magnification for evidence of poor adhesion. Any peel or flaking from the basis metal was arbitrarily considered a failure. Plate thickness was 2 mils.

C. Porosity

Specimens were exposed to 20% salt spray as described in Federal Test Method Standard 151, Method 811, until failure or completion of a 250 hour test. The first appearance of rust was considered a failure.

D. Hardness

Hardness tests were conducted on a Wilson-Tukon hardness tester using a Knoop indenter. The load was 100 grams and the lens was 4.0 with a factor of .1662.

The tests were conducted on a 3 mil plating applied to 4130 steel. Specimens were sawed, mounted and tested in a position perpendicular to saw direction so that indenter impinged only into the chromium.

E. Crack Detection

A specimen of 4130 steel was plated with conventional chromium while another was plated with Crack-free 500 chromium. Both were plated with .002" thickness chromium. The specimens were then examined on a metallograph at various magnifications; however, a magnification of 250 X was best suited for crack detection.

F. Sustained Load Tests

In order to arrive at a 90% ultimate load for actual sustained load tests standard sheet tensile specimens, taken in the longitudinal direction, were fabricated according to drawing FTJ-10940-8.* Standard notched sheet tensile specimens, also taken in the longitudinal direction, were fabricated as shown in drawing FTJ-10940-20.* After fabrication specimens were heat treated as follows:

1. Preheat to 1450°F - hold for 30 minutes (argon atmosphere)

2. Heat to 1850°F - hold for 1 hour (argon atmosphere)

3. Air cool

4. Double temper at 1025°F for 2 hours (argon atmosphere).

The tensile tests for both notched and unnotched specimens were conducted in a 60,000 pound Baldwin universal test machine. A microformer extensometer and a MA-1 stress-strain recorder were used to obtain the yield strength by the 0.2% offset method on the unnotched specimens. Ninety percent of the average notched tensile specimen value was used as load for sustained load tests.

Specimens for sustained load were additional notched tensile specimens, fabricated, heat treated, sanded, polished, plated and baked as shown in Table IV. Sustained load tests were run on an Arcweld 12,000 pound creep-rupture machine until failure or completion of 1000 hour test.

*See Supplemental Sheet S-1

After sustained load tests the notched area was removed with an alundum water cooled cut off saw. The areas removed were .055" x 0.25" x 0.25". Specimens were then thoroughly cleaned in acetone, dried and analyzed in the vacuum hydrogen analyzer. A few additional specimens were examined on the outer edge as shown in Table IV.

G. Hydrogen pick-up determinations*.

Three strips approximately 6" long x 1/4" wide of .055" Thermold A material were sheared out of sheet stock. One strip was used as unplated control, one strip was plated with .002" conventional chromium and the remaining strip was plated with .002" of Crack-free 500 chromium. The strips were identified and sheared into pieces 1/4" x 1/4" x .040". Specimens were baked out as groups at various conditions of time and temperature shown in Table V. Hydrogen content of specimens after various treatments was determined on a N.R.C. vacuum hydrogen determinator according to procedures described in FZM-1776, procedure 3.560.

RESULTS:

Figure 1 is a photomicrograph at 250 X magnification of Crack-free-500 chromium and conventional chromium. Table I presents data for visual observations, adhesion and hardness tests. Salt spray test data are shown in Table II while Table III and IV contain results of tensile, sustained load and hydrogen content of sustained load tests. Effects of various bake-out conditions on hydrogen content are shown in Table V.

DISCUSSION:

Upon examination of Figure 1 it is evident that Crack-free 500 chromium can be deposited in the crack-free state. Table I data shows the two materials to be comparable except in hardness with Crack-free 500 chromium being somewhat harder. Salt spray test data from Table II is conclusive that Crack-free 500 is considerably more resistant to corrosion. For instance .003" conventional chromium failed 48 hours salt spray while the same thickness Crack-free 500 chromium passed 250 hours without any basis steel corrosion. This is also further proof that the Crack-free 500 is crack-free.

*See Supplemental Sheet S-2

Sustained load test data in Table IV shows first that 375°F for 23 hours bake-out of Crack-free-500 is not sufficient hydrogen relief since all specimens failed the 1000 hour test. With more bake out, for instance 500°F for 2 hours, adequate sustained load strength was obtained. Data for conventional chromium sustained load tests agrees with published data which indicates 375°F for 23 hours is an adequate bake-out condition. This data therefore is good control data. It is also obvious, generally speaking, that the analyzed hydrogen content is less at more elevated conditions of temperature and time. Some difficulties were encountered in the hydrogen determination tests. During cut-off operations with the alundum wheel visible oxidation occurred on the specimens even though they were cut under water. For this reason it was decided to conduct additional tests for hydrogen determinations with emphasis on eliminating the cut-off heating problem. Thin strips of metal were plated and small specimens were then sheared off for various bake-out conditions and subsequent hydrogen analysis. By examining data from these tests reported in Table V it is obvious that the hydrogen content is reduced by increasing the bake out time and temperature.

CONCLUSIONS:

The chemical and physical properties, hydrogen embrittlement effects and methods of hydrogen embrittlement relief for Crack-free-500 were investigated. The following conclusions are made:

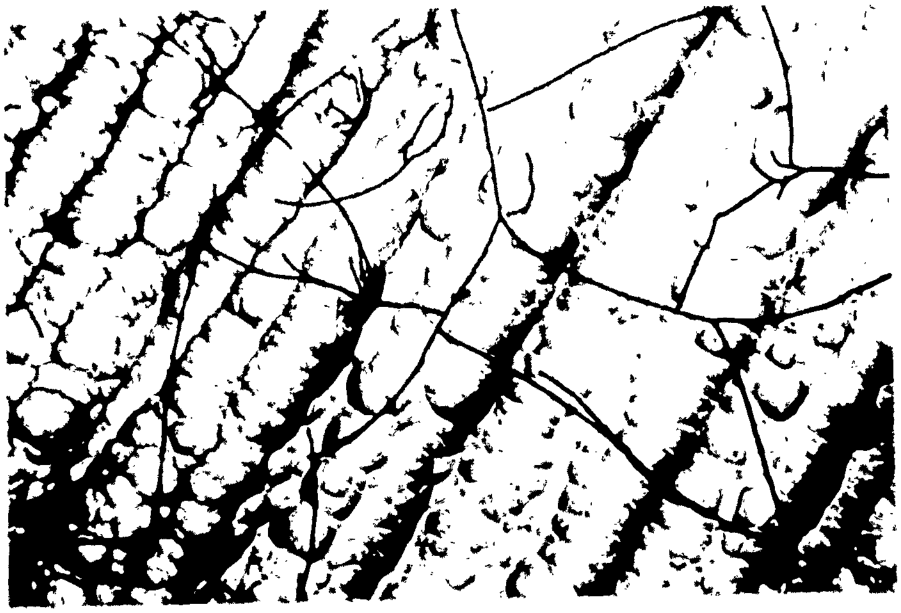
1. CF-500 chromium is crack free.

2. CF-500 chromium plated steel is more resistant to corrosion than conventional chromium plated steel.

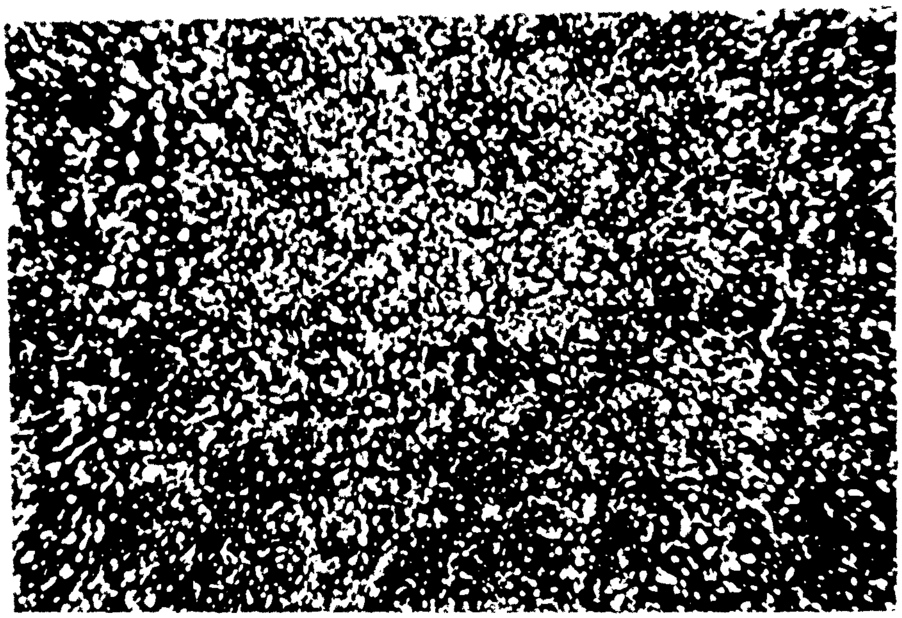
3. CF-500 chromium is comparable to conventional chromium in appearance and adhesion but is slightly harder than conventional chromium plate.

4. Adequate hydrogen embrittlement relief can be obtained from CF-500 by increasing bake out time and temperature to 500°F for 23 hours.

FIGURE 1



CONVENTIONAL CHROMIUM .002" THICK 250X



CRACK-FREE 50% CHROMIUM .002" THICK 250X

TABLE I
TEST RESULTS

I. VISUAL OBSERVATIONS

TYPE OF PLATING	PITS	BLISTERS	EDGE BUILD-UP	SURFACE APPEARANCE
CONVENTIONAL CHROMIUM	NONE	NONE	SLIGHT	SMOOTH, GREY, MATTE
CRACK-FREE 500	"	"	"	" " "

II. ADHESION - (4X MAGNIFICATION) - BEND TEST

CONVENTIONAL CHROMIUM	CRACK-FREE 500
PASS	PASS

III. HARDNESS - TUKON HARDNESS TESTER USING KNOOP INDENTER

CONVENTIONAL CHROMIUM

	KNOOP	ROCKWELL C*
	889	70+
	847	67.6
	889	70+
	847	67.6
	770	63.5
	593	53.5
AVG.	806	65.3+

CRACK-FREE 500

	KNOOP	ROCKWELL C*
	847	67.6
	1039	70+
	936	70+
	889	70+
	985	70+
	936	70+
AVG.	939	69.6+

* SCALE CONVERSION FROM KNOOP DETERMINATION

TABLE II

EFFECTS OF 250 HOUR SALT SPRAY EXPOSURE PER FEDERAL TEST METHOD STANDARD 151, METHOD 811

BASIS METAL 4130 FAILURE DEFINED AS 1ST APPEARANCE OF RUST

TYPE OF PLATING	THICKNESS, INCHES	RESULTS OF SALT SPRAY TESTS
CONVENTIONAL CHROMIUM 53 oz/GAL	0.0005	PASSED 250 HOURS (1 SPECIMEN)
"	0.001	3 OF 6 FAILED AT 8 HOURS, 1 FAILED 24, 1 FAILED 72, 1 PASS 250 HOURS
"	0.002	2 OF 6 FAILED 24 HOURS, 3 FAILED 48 HOURS AND ONE FAILED AT 96 HOURS
"	0.003	3 OF 6 FAILED 24 HOURS, 3 FAILED 48 HOURS
CRACK-FREE 500	0.0005	FAILED 216 HOURS, ONE SPECIMEN
"	0.001	3 OF 3 FAILED AT 104 HOURS
"	0.002	3 OF 3 PASSED 250 HOURS
"	0.003	2 OF 2 PASSED 250 HOURS

TABLE III

STANDARD TENSILE AND NOTCHED TENSILE TEST DATA FOR THERMOLD A STEEL

TYPE TEST	GAUGE INCHES	WIDTH INCHES	AREA SQ. INCHES	YIELD POINT POUNDS	YIELD POINT K.S.I.	ULTIMATE POUNDS	ULTIMATE K.S.I.	PERCENT ELONGATION
STANDARD 1.	.0555	.4117	.0228	5070	221.9	5870	256.9	6
TENSILE 2.	.0530	.4150	.022	4660	211.8	5330	242.4	5
AVG.	—	—	—	—	208.8	—	249.6	—
STANDARD	.0581	.440	.0255	—	—	5910	231.2	—
NOTCHED TENSILE	.0541	.435	.0235	—	—	5080	216.2	—
AVG.							223.7	

TABLE IV

RESULTS OF HYDROGEN ANALYSIS AND SUSTAINED LOAD TESTS ON CONVENTIONAL AND CRACK-FREE CHROMIUM PLATED* THERMOID "A" STEEL

PLATING PROCESS	SPECIMEN No.	BAKE-OUT CONDITION AFTER PLATING TEMP. °F	TIME HOURS	HOURS TO FAILURE AT 200 KSI STRESS	PARTS PER MILLION HYDROGEN AT NOTCH	AT OUTER EDGE	REMARKS
CF-500 CRACK-FREE CHROMIUM	1	NONE	NONE	0	35.0	—	FAILED ON LOADING
	2	"	"	0	33.5	—	" " "
	3	"	"	0	28.5	—	" " "
	4	"	"	0	30.0	—	" " "
	5	375	23	0	29.0	—	" " "
	6	"	"	0	33.0	—	" " "
	7	"	"	0	31.5	—	" " "
	8	"	"	389.9	26.0	—	FAILED 1000 HR. TEST
	9	"	"	0	19.5	—	FAILED ON LOADING
	10	"	"	0	29.5	—	" " "
	11	500	23	1533.3 →	—	—	PASSED 1000 HR. TEST
	12	"	"	1533.2 →	—	—	" " " "
	13	"	2	1122.8 →	—	—	" " " "
	14	"	"	1127.7 →	—	—	" " " "
	15	625	23	1533.6 →	—	—	" " " "
	16	"	"	79.9	—	—	SPECIMEN HAD VISIBLE FLAW
	17	"	2	1145.0 →	—	—	PASSED 1000 HR. TEST
	18	"	"	1145.0 →	—	—	" " " "
	19	750	24	1506.4 →	—	—	" " " "
	20	"	"	825.3	—	—	SPECIMEN HAD VISIBLE FLAW
	21	1000	2	1027.3 →	22.0	—	PASSED 1000 HR. TEST
	22	"	"	0	17.0	—	HELD ONLY 10 SECONDS
	23	"	"	1026.3 →	34.0	—	PASSED 1000 HR. TEST
	24	"	"	1026.4 →	22.0	—	" " " "
	25	"	"	1053.1 →	23.0	—	" " " "
	26	"	"	1053.2 →	24.0	—	" " " "
CONVENTIONAL (53 oz/GAL) CHROMIUM	27	NONE	NONE	0	50.0	—	FAILED ON LOADING
	28	"	"	0	36.5	—	" " "
	29	"	"	0	40.5	—	" " "
	30	"	"	0	36.5	—	" " "
	31	1000	2	1441.3 →	33.0	31.8	PASSED 1000 HR. TEST.
	32	"	"	1002.5 →	25.0	26.0	" " " "
	33	"	"	1050.1 →	26.0	31.1	" " " "
	34	"	"	0	29.0	33.6	FAILED ON LOADING
	35	"	"	1050.0 →	24.0	31.5	PASSED 1000 HR. TEST
	36	"	"	1081.8 →	27.0	—	" " " "
	37	375	23	0	36.5	—	FAILED ON LOADING
	38	"	"	1012.2 →	26.0	—	PASSED 1000 HR. TEST
	39	"	"	—	—	—	SPECIMEN LOST
	40	"	"	1008.6	22.0	—	PASSED 1000 HR. TEST
	41	"	"	1278.5 →	36.0	—	" " " "
	42	"	"	1277.3 →	50.0	—	" " " "

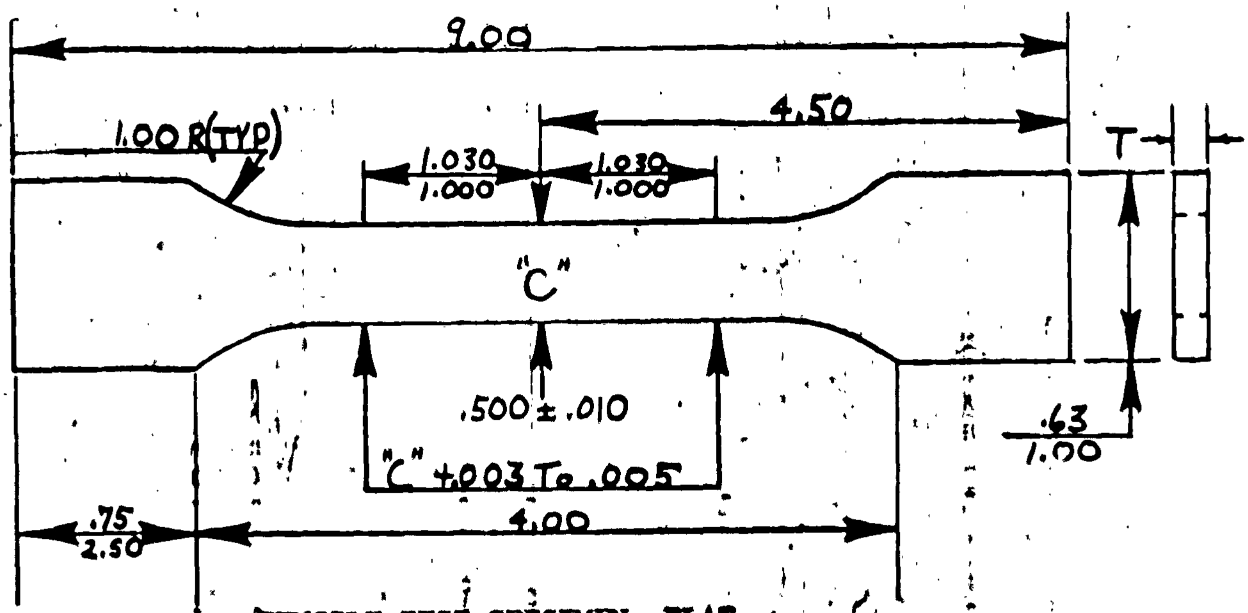
→ DENOTES SPECIMENS DID NOT FAIL BUT WERE REMOVED AFTER TIME SHOWN

UTILITY REPORT SHEET *THICKNESS .002" SLIGHT OXIDATION AFTER EXPOSURE

TABLE V

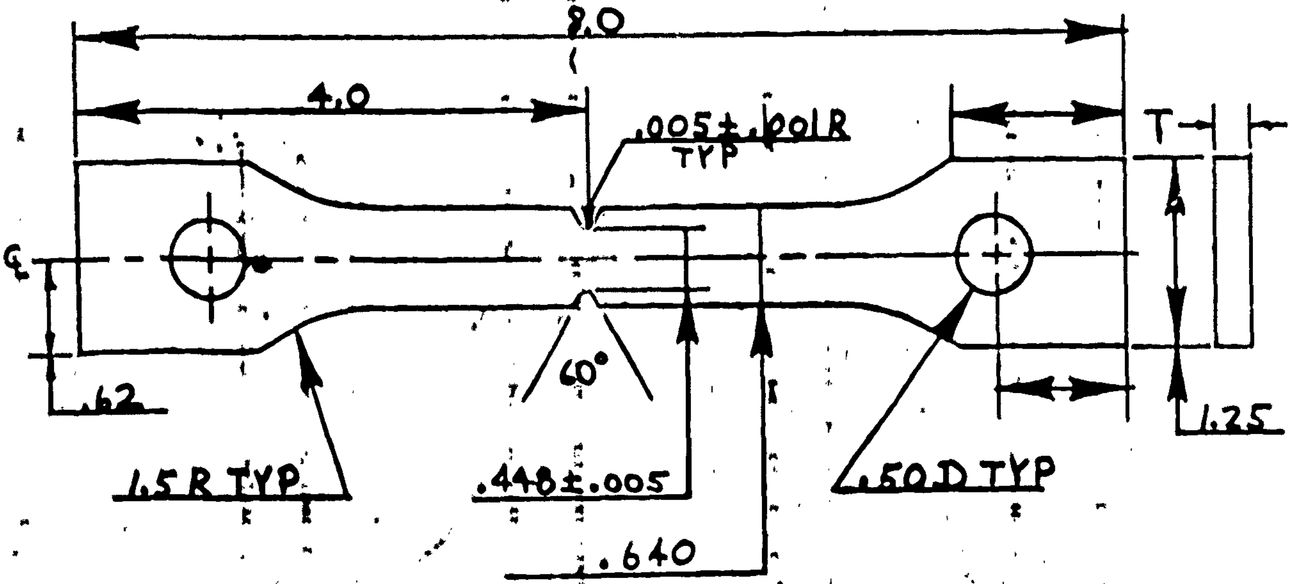
EFFECTS OF VARIOUS BAKE-OUT CONDITIONS ON HYDROGEN CONTENT OF PLATED THERMOLD A. STEEL

SPECIMEN NUMBER	TYPE OF PLATING	BAKE OUT CONDITION °F	BAKE OUT CONDITION HOURS	PARTS PER MILLION HYDROGEN	SPECIMEN NUMBER	TYPE OF PLATING	BAKE-OUT CONDITION °F	BAKE-OUT CONDITION HOURS	PARTS PER MILLION HYDROGEN
1	NONE	NONE	NONE	11.7	21	CRACK-	NONE	NONE	71.5
2	"	"	"	13.9	22	FREE 500	"	"	68.3
3	"	"	"	21.3	23		"	"	64.0
4	"	"	"	16.9	24		"	"	71.4
5	"	"	"	19.5	25		"	"	72.2
AVG	"	"	"	16.7	AVG		"	"	69.5
6	CONVENTIONAL	"	"	8.6	26	CRACK-	1000	2	33.0
7	CHROMIUM	"	"	7.8	27	FREE	"	"	30.3
8	53 g/GAL	"	"	8.3	28	500	"	"	31.4
9		"	"	11.7	29		"	"	29.6
10		"	"	36.5	30		"	"	30.7
AVG				14.5	AVG		"	"	31.0
11	CONV.	1000	2	15.1	31	CRACK-	375°F	23	12.8
12	CHROMIUM	"	"	11.0	32	FREE	"	"	8.6
13		"	"	9.5	33	500	"	"	10.1
14		"	"	15.5	34		"	"	9.8
15		"	"	9.8	35		"	"	9.7
AVG		"	"	12.1	AVG		"	"	10.2
16	CONV.	375°F	23	3.1					
17	CHROMIUM	"	"	40.8					
18		"	"	4.6					
19		"	"	12.0					
20		"	"	10.3					
AVG		"	"	14.2					



TENSILE TEST SPECIMEN--FLAT

FTJ-10940-8



NOTCHED SHEET TENSILE SPECIMEN

FTJ-10940-20

Hydrogen Determination, Reference Page 7, Paragraph G

Hydrogen content of the metal was determined by the hot vacuum extraction method. A National Research Corporation Vacuum Determinator, Model 917, was used.

Metal samples were placed in a molybdenum crucible and heated by an induction furnace. At elevated temperatures the sample releases the contained hydrogen which is pumped into a measured volume. The change in pressure of the measured volume is indicated by a Mcleod gauge. The hydrogen content in parts per million is calculated as follows:

$$(H_2, \text{ppm}) = \frac{(0.1084,\ \text{Volume Constant})(\text{Volume})(\text{Pressure, Microns})}{(\text{Weight of Sample, Grams})}$$